

# Effect of High Temperature Storage in Vacuum, Air, and Humid Conditions on Degradation of Gold/Aluminum Wire Bonds in PEMs

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## Abstract

*Microcircuits encapsulated in three plastic package styles were stored in different environments at temperatures varying from 130°C to 225°C for up to 4,000 hours in some cases. To assess the effect of oxygen, the parts were aged at high temperatures in air and in vacuum chambers. The effect of humidity was evaluated during long-term highly accelerated temperature and humidity stress testing (HAST) at temperatures of 130°C and 150°C. High-temperature storage testing of decapsulated microcircuits in air, vacuum, and HAST chambers was carried out to evaluate the role of molding compounds in the environmentally-induced degradation and failure of wire bonds (WB). This paper reports on accelerating factors of environment and molding compound on WB failures. It has been shown that all environments, including oxygen, moisture, and the presence of molding compounds reduce time-to-failures compared to unencapsulated devices in vacuum conditions. The mechanism of the environmental effect on WB degradation is discussed.*

*Key words: wire bond, PEM, reliability, environment.*

## Introduction

A substantial proportion of package-related failures in plastic encapsulated microcircuits (PEMs) is caused by failures of gold/aluminum wire bonds (WBs) and this degradation is one of the major factors limiting reliability of the parts at high temperatures. In order to qualify PEMs for the harsh-environments of military and space applications, a comprehensive evaluation of the effect of environments on reliability of WBs is required. Up to date, only a limited number of studies have addressed these issues. Hydrogen was found to inhibit formation of Au/Al intermetallic (IM) compounds during high temperature annealing at 400°C [1]. Moisture significantly reduces time to WB failures by enhancing transportation to the bonds of halogen molecules which break from the flame retardants [2] or working as a catalyst [1]. The effect of oxygen can be due to accelerated decomposition of MC, which increases the flow of halogens to WBs, or to a direct oxidative degradation of Au/Al IMs [3]. Vacuum conditions might be beneficial to reliable operation of PEMs; however, the significance of this effect depends on the integrity of the package and the possibility of thermo-oxidative decomposition of MC in the vicinity of the bonds [3, 4]. Besides, the role of MC in degradation of WBs in vacuum has not been studied yet.

The data reported in literature on activation energies of WB failures vary in a wide range from 0.3 to 2.5 eV. This is partially due to different definitions of failure and different techniques used by various authors. For example, in some cases the time to a WB failure is assumed to be equal to the time of a full growth of the IM [5]. However, at 175°C aluminum is typically fully consumed from contact pads under the bonds to form IMs in ~ 10 hrs [6] and their growth stops in a few dozens of hours only, whereas normal quality bonds do not fail after hundreds and thousands of hours of high temperature annealing even above 175°C [7, 8].

Due to the higher resistivity of Au/Al IMs compared to Au and Al, their growth increases contact resistance,  $R_c$ , from 10 to 80 mOhm only [9-11], which is negligible in most applications and not considered as a reliability hazard [1]. Typically, electrical failures of microcircuits occur when  $R_c$  increases to more than 1 Ohm [12, 13].

In this work measurements of contact resistance were employed to non-destructively monitor the degradation processes in wire bonds in commercial PEMs during high temperature storage (HTS) testing at different temperatures in air, vacuum, and humid environments. To evaluate the role of MC, experiments in different environments were performed on decapsulated microcircuits along with encapsulated devices.

## Experiment

Three groups of commercial low-voltage CMOS analog devices in different plastic packages have been used for this study. The first group, EEE, was encapsulated in 16-QSOP packages with orthocresol novolac (OCN) epoxy MC, the second, EUE, was encapsulated in 16-TSSOP with Bi-phenyl (BP) MC, and the third, EPE, was encapsulated in a plastic 16-DIP with OCN MC. Two groups of EEE devices, EEE I and EEE II, with different date codes (manufactured ~ 4 years apart) were used and tested at different temperatures to evaluate the consistency of the results.

X-ray microanalysis was used to evaluate concentrations of Br and Sb in molding compounds. Results of these measurements are shown in Table 1.

Table 1. Characteristics of molding compounds

Part	MC type	Br, wt. %	Sb, wt. %
EEE	OCN	0.6	0.7
EUE	Bi-phenyl	0.4	0.5
EPE	OCN	1.2	2.6

HTS testing was performed in air chambers at temperatures varying from 175 to 225 °C for up to 2,000 hours and in a vacuum chamber at 198 °C for 2,400 hours. Highly accelerated stress testing, HAST, was carried out at 85% RH and temperatures of 130 °C for 4,400 hours and 150 °C for 3,100 hours. To assure that the parts stored in air and vacuum conditions had the same temperature (198 °C), a small vacuum cell with a pressure maintained at ~0.1 torr, was placed in a regular air chamber next to the air-stored parts. The temperature during HTS was monitored directly in the area of the parts' location.

Each group of parts had 10 to 15 samples and  $R_c$  values were estimated at a current of 5 mA using a forward voltage drop technique [14]. A bond was considered as failing catastrophically when  $R_c$  increased to 10 Ohm. A Weibull function was used to evaluate parameters of distributions of WB failures. HTS testing of decapsulated devices was performed after MC had been removed from the die surface by jet etching using red fume nitric acid, rinsing with acetone, and dry air blowing.

## Results

### Effect of temperature in air.

Distributions of WB failures for EEE devices are plotted in Weibull coordinates in Fig. 1. At temperatures in the range from 190 to 200 °C most WB failures were observed between 200 and 1000 hrs, which agrees with the results of Iwasaki and

Ueda [8] where an increase of  $R_c$  to 10 Ohm was observed after ~700 hrs of annealing at 185 °C, and with the data obtained by of Uno et al. [15], where analogous  $R_c$  variations were detected after ~400 hrs at 200 °C. Slope of the distributions tends to increase as temperature decreases (from 2.9 at 200 °C to 5.5 at 175 °C). This trend has been observed also in [4, 15].

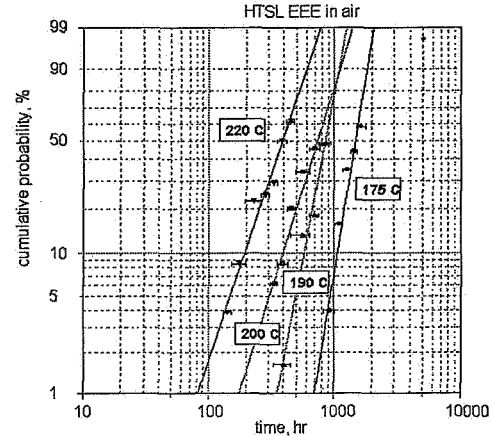


Figure 1. Distributions of WB failures in EEE devices in air at different temperatures.

Values of the characteristic life time,  $\eta$ , and slope,  $\beta$ , of the distributions were calculated for each group of devices and temperature dependencies of  $\eta$  for parts in different packages are shown in Fig. 2. For different part types  $\eta$  at 200 °C varied in a relatively narrow range, from 500 to 1000 hrs. The times to failure for EPE devices were lower than for EEE parts probably due to a higher level of contamination in MC and the larger thickness of package. Notably, two lots of EEE devices manufactured with a four-year interval had similar results.

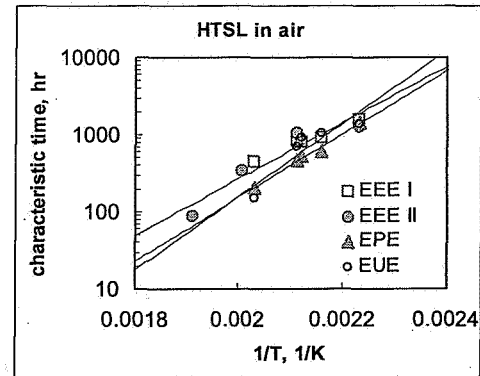


Figure 2. Temperature dependencies of characteristic life times of WBs for different part types.

Based on these results activation energies,  $E_a$ , of WB failures were calculated as 0.71 eV, 0.91 eV, and 0.79 eV for EEE, EUE, and EPE devices respectively. For devices in BP MC (EUE)  $E_a$  is

close to what has been reported by Biddle [16] (from 1 to 1.1 eV), but much lower than 1.5 eV reported by Uno and Tatsumi [15] for a temperature range between 185 and 200 °C.

In all cases of failures, lifted bonds with a grainy surface of IMs were revealed after decapsulation (see Fig. 3). EDS microanalysis showed that the grains were gold-rich compositions, likely  $\text{Au}_8\text{Al}_3$  or  $\text{Au}_4\text{Al}$ . The size of the grains was 200 to 400 nm and in some cases a lamellar microstructure with a size of  $\sim 20$  nm was observed (Fig. 3c). Intergranular holes and pits in the grains were formed likely by a dry corrosion process.

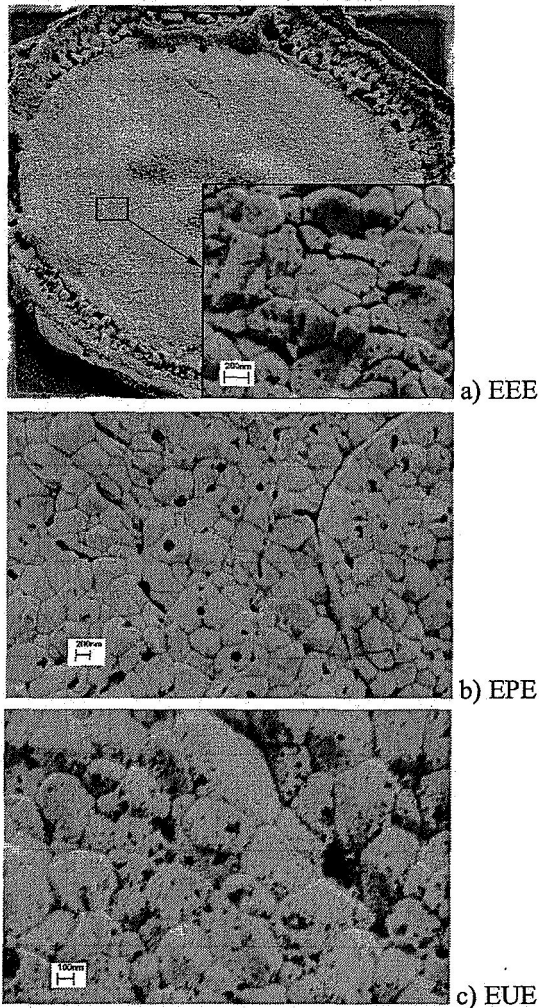


Figure 3. Morphology of the surface of Au/Al intermetallics in different parts after HTS testing at 175 °C for 1800 hrs.

#### Effect of vacuum

Table 2 gives a comparison of the characteristic times to failure for encapsulated devices stored at 198 °C in air and vacuum conditions. The data indicate a relatively small

increase of  $\eta$  in vacuum compared to air conditions. For devices encapsulated in OCN MC this increase was from 25% to 35%, similar to what was observed earlier for microcircuits encapsulated in QFP packages [4]. Interestingly, there was no significant change in  $\eta$  for devices encapsulated with BP MC.

Table 2. Effect of vacuum on characteristic life, hr, of WBs at 198 °C.

Part	Air	Vacuum	ratio
EEE	793	1010	1.25
EUE	903	951	1.05
EPE	529	717	1.35

Distributions of WB failures at 198 °C for decapsulated parts stored in air are shown in Fig. 4. If aluminum metallization and gold wire materials used in parts encapsulated in different packages were the same, one might expect similar results for all types of decapsulated devices. The slopes of the distributions were close and varied from 2.2 for EPE devices to 3 for EUE parts indicating a similar mechanism of failures. Also, test results for EEE and EUE parts were similar; however, times-to-failure in decapsulated EPE devices were approximately two times less, which might be related to some differences in the metallization system used in EPE devices, which were manufactured much earlier than other parts. The lesser values of  $\eta$  for decapsulated EPE devices compared to decapsulated EEE parts suggest that the observed difference (Fig. 2) in  $\eta$  for PEMs encapsulated in OCN MCs might be due not only to the difference in concentration of Br and As, but also to the difference in the metal systems used.

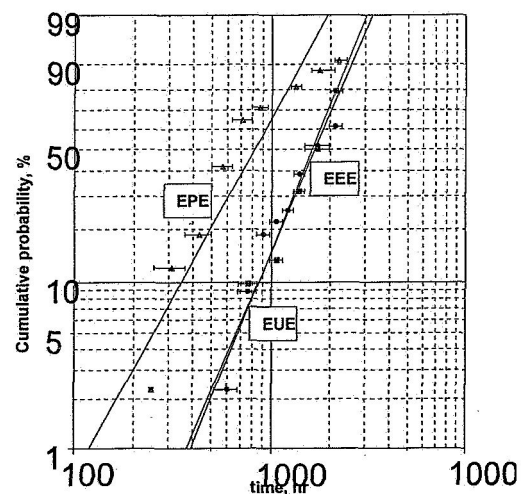


Figure 4. Distribution of WB failures during HTS testing of decapsulated parts in air at 198 °C.

A comparison between the characteristic life times of WBs in air at 198 °C for encapsulated and decapsulated parts is shown in Table 3. In all cases removal of MC approximately doubles  $\eta$ . This result agrees with the data reported in [17] where HTS testing at 200 °C revealed a substantial decrease in corrosion of Au/Al IMs in decapsulated parts compared to encapsulated devices.

Table 3. Characteristic life of WBs in air at 198 °C for encapsulated and decapsulated microcircuits.

Part	$\eta_{\text{decap.}}$ , hr	$\eta_{\text{encap.}}$ , hr	Ratio
EEE	1832	793	2.3
EUE	1903	903	2.1
EPE	971	529	1.8

Decapsulated EPE devices were tested in vacuum at 198 °C and results of this testing together with the results of air storage are shown in Fig. 5.

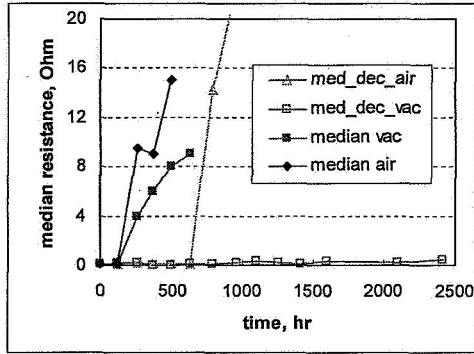


Figure 5. Variations of contact resistances with time of storing at 198 °C for encapsulated and decapsulated EPE devices in air and vacuum environments.

Although decapsulated EPE devices were found to have a metallization system with the highest susceptibility to WB degradation and first failures in air were already observed after ~ 250 hrs, no increase in  $R_c$  was detected even after 2500 hrs of annealing in vacuum. This data indicated a substantial increase in time to WB failures for decapsulated devices in vacuum compared to encapsulated devices in vacuum and/or decapsulated devices in air environments.

Wire pull testing of decapsulated EPE devices after HTS at 198 °C for 2400 hrs showed that all parts stored in air environments were separated along the IMs and 95% of bonds had a pull force of less than 2 g. The proportion of WB pull test failures (<2 g) for decapsulated parts stored in vacuum, was ~30% only and ~ 25% of WBs were broken at the neck at a pull force of more than 5 g. All WBs in decapsulated parts aged in vacuum and which failed pull test, exhibited a characteristic appearance of fracture and

broke along the intermetallics/barrier metal interface (see Fig. 6). These bonds had extensive IM growth along the periphery of the bond and were separated from the die at the central area and along the peripheral bump.

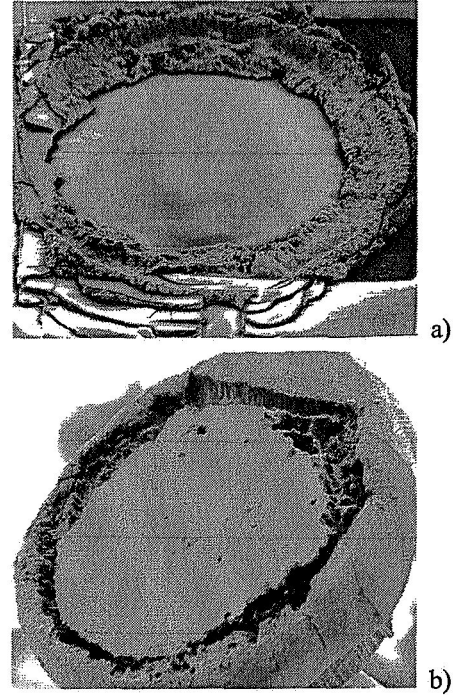
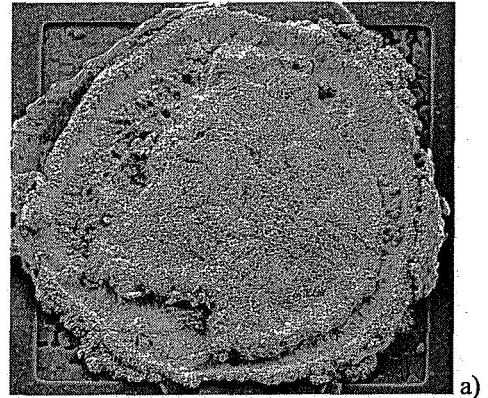


Figure 6. WBs after pull testing in decapsulated EPE devices stored in vacuum at 198 °C for 2400 hrs.

In contrast, WB failures in decapsulated parts after air storage separated along the IMs as shown in Fig 7. Intergranular voiding at the corners of aluminum contact pads observed in these as well as in most of other HTS experiments suggests that diffusion of Al atoms occurs mostly along the grain boundaries of aluminum metallization. A close-up view indicates the different morphology of the surface compared to encapsulated devices (Fig. 3).



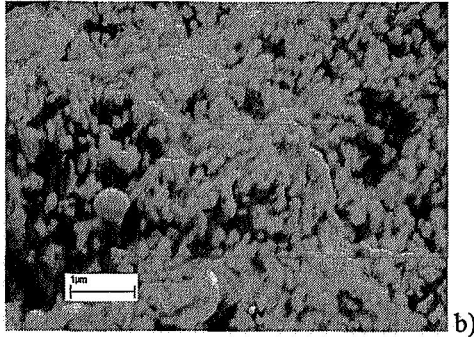


Figure 7. Overall (a) and close-up (b) views of a failed WB in decapsulated EPE devices stored in air at 198 °C for 2400 hrs.

#### Effect of humidity

Storing in humid environments at 85 %RH and 130 °C during 4000 hrs did not result in WB failures in parts encapsulated with OCN MC (EEE and EPE). However, at 150 °C/85 %RH, the first failures of these devices were observed after ~ 1200 hrs of testing. In parts encapsulated with BP MC (EUE) the rate of failure was much higher and first failures at 130 °C/85 %RH were observed after 1500 hrs, and at 150 °C/85 %RH the WBs started failing already after ~80 hrs. A typical view of the failed WBs encapsulated in BP MC is shown in Fig. 8. No corrosion of Al contact pads or IM growth was observed along the periphery of these bonds. The morphology of intermetallics (Fig. 8c) indicates a grainy structure of a Au-rich composition with grain sizes of ~ 100 to 200 nm, similar to the air stored decapsulated devices (Fig. 7).

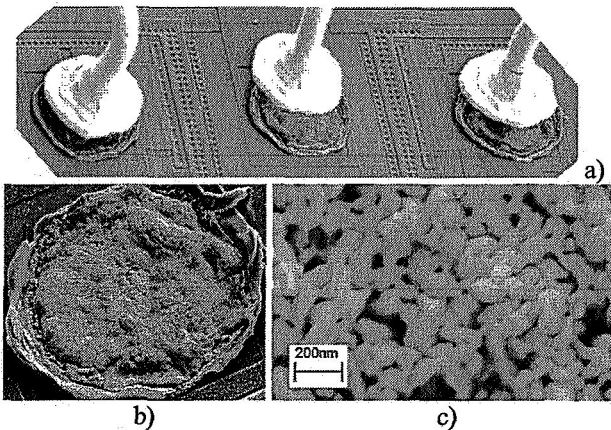


Figure 8. Lifted wire bonds in EUE devices after HAST at 150 °C/85 %RH for 4000 hr (a, b) and a close-up of the central area (c).

For encapsulated EUE devices, distributions of HAST failures are shown in Fig. 9. The slopes of distributions in humid environments were close (2.9 at 130 °C and 2.1 at 150 °C) indicating similar failure mechanisms. Fig. 9 also shows distributions of times

to failure for EUE devices in dry conditions at 130 °C and 150 °C, calculated based on extrapolation of temperature dependencies of  $\eta$  and  $\beta$  from the range of 175 to 225 °C to lower temperatures. A steeper slope of distributions in dry conditions (8.6 to 9) might suggest a different mechanism of failures.

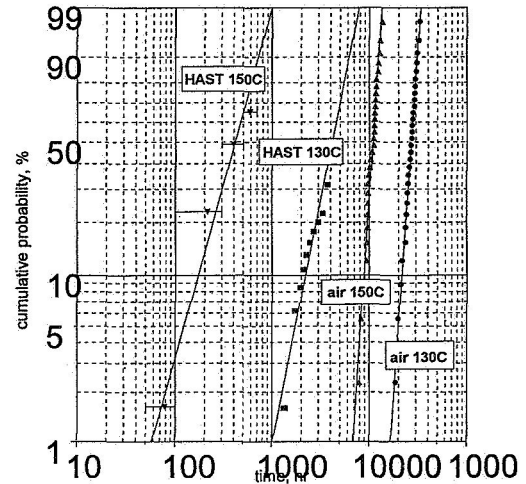


Figure 9. Distributions of WB failures at 130 °C and 150 °C in humid (85 %RH) and dry air environments.

The results show that for devices in BP MC moisture reduces the characteristic time of WB failures compared to dry air conditions by a factor of ~20 at 150 °C and ~ 6 at 130 °C. Similar calculations yielded lower moisture acceleration factors for OCN MC varying from 2.3 to 4 at 150 °C. BP MCs are considered as less thermally stable allowing higher rates of liberation and transport of halogens to WBs and, respectively, causing higher failure rates of WBs compared to OCN materials [8, 15]. Our data indicate that BP MCs is inferior compared to OCN materials in humid environments only and have similar failure rates in dry conditions.

Table 4 shows characteristic times of WB failures and activation energies in humid environments. In the absence of WB failures for devices in OCN MCs at 130 °C/85% RH, only estimations of the minimal level of  $E_a$  could be made. Based on these estimations,  $E_a$  exceeds 0.7 and 1 eV for EEE and EPE parts respectively.

Table 4. Effect of temperature at 85 %RH on characteristic life, hr, of WB failures.

Part	130 °C	150 °C	$E_a$ , eV
EEE	*	2640	>0.7
EUE	4715	490	1.6
EPE	*	2380	>1

\* no failures after 3940 hrs of testing



Two groups of decapsulated devices (EUE and EPE) were tested in the HAST chamber at 150 °C/85 %RH and no WB failures were observed up to 2400 hrs of testing. This suggests that similar to air conditions, the presence of MC significantly increases the rate of WB failures in humid environments also. At 150 °C/85 %RH a decrease of the time to WB failures caused by MC is more than 40 times for BP and more than 2.5 times for OCN epoxies.

## Discussion

The most important results of this investigation are summarized in Tables 5 and 6 where estimations of the characteristic life of WBs in different environments are displayed. In the case where no failures were observed, the values of  $\eta$  were estimated assuming that the first failure occurred at the end of the test.

Table 5. Characteristic life, khr, of WBs in different environments at 198 °C

Encapsulant	Vacuum	Air
Decap.	>(10 - 14)	1 - 1.8
OCN	0.7 - 1	0.5 - 0.8
BP	0.95	0.9

Table 6. Characteristic life, khr, of WBs in humid (85 %RH) and dry conditions at 150 °C

Encapsulant	Moisture	Air
Decap.	>(7 - 10)	>(20-40)*
OCN	2.4 - 2.6	6 - 9
BP	0.5	11

\* assuming  $E_a > 1$  eV

The highest reliability of WBs during HTS testing has been demonstrated for decapsulated devices in vacuum. Considering that the degradation of WBs in PEMs is mostly due to low molecular weight corrosive species liberated from epoxies at high temperatures, the presence of MC can be considered as an environmental factor also. In this contest, our results indicate that all environments, including oxygen, moisture, and molding compounds significantly accelerate degradation of Au/Al wire bonds compared to vacuum conditions.

The evolution of Au/Al IMs during high temperature annealing has been studied by many authors [1, 11, 18, 19]. It has been shown that after a long-term aging at high temperatures the central, disk-like part of the bond is transformed into a gold-rich phase,  $Au_4Al$ , adjacent to  $Au_8Al_3$  phase at the bottom and Au ball at the top. The  $Au_4Al/Au_8Al_3$  interface has microvoids created as a result of the

Kirkendall effect and/or volumetric changes in the composites and their concentration is typically larger along the ball edges. Further exposure to high temperatures converts  $Au_8Al_3$  into  $Au_4Al$  thus increasing the thickness of the gold-rich phase. Due to abundance of aluminum atoms along the periphery of the bond, reverse transformations might occur with re-formation of  $Au_8Al_3$  areas under the bump with microvoids and creep cavities accumulated along the interfaces. Volumetric changes associated with these transformations create mechanical stresses pushing the ball up, might cause formation of interfacial cracks, and enhance separation of the center part of the ball surface from the chip during pull testing. This explains the appearance of failures in vacuum-stored decapsulated parts (see Fig. 6).

For decapsulated devices the presence of air significantly reduces, by a factor of >10, time to WB failure, which indicates an important role of oxidation processes in the degradation of intermetallics. It is known that  $Au_4Al$  composition is prone to corrosion more readily than other Au/Al IMs and decomposes rapidly in the presence of halides [20]. It has been shown recently that appreciable amounts of oxidation can occur in gold aluminides,  $AuAl_2$  and  $Au_4Al$  [21] and the latter has higher oxidation rates than Al and  $AuAl_2$ . During oxidation, Al atoms diffuse out of IMs to form  $Al_2O_3$  at the surface, leaving a gold-enriched phase beneath the oxide. It is reasonable to assume that oxidation occurs mostly along the areas with microvoids and goes along with the local volumetric changes. In this case, microcracks along the  $Au_4Al/Au_8Al_3$  interface would form and further facilitate the oxidation process. Eventually, this process increases contact resistance and the possibility of fracture of WBs. It is quite possible that moisture can react with Au/Al IMs in a similar way accelerating WB failures.

The present data as well as our previous studies [3, 4] show that vacuum conditions as experienced in space applications are beneficial for PEMs. However, the effect is not very strong varying from 3-8 times for devices with surface delaminations to 25% - 35% for parts without delaminations. A relatively weak effect of vacuum conditions in PEMs is most likely due to thermal decomposition of the MCs prevailing over thermo-oxidative decomposition and to outgassing of MCs, which provides sufficiently large flow of oxidative molecules to oxidize Au-rich IMs. It is known that at high temperatures epoxy encapsulants can release a considerable amount of water and  $CO_2$  [22] and outgassing products of epoxy adhesives substantially increase the growth of  $Al_2O_3$  on aluminum contact pads [23].

The presence of MCs significantly reduces the characteristic life of WBs, which is commonly explained by the presence of bromide causing dry corrosion of Au-rich aluminides (formation of aluminum bromide and its oxidation to  $\text{Al}_2\text{O}_3$ ). One might expect that transferring to green, bromide-free, MCs would have substantially improved the reliability of bonds. However, it has been shown recently that significant degradation of Au/Al WBs might occur even with bromide-free MCs [24]. Our results suggest that oxidative molecules liberated from MC at high temperatures play an important role in the degradation process causing conversion of gold-rich IMs into aluminum oxide resulting in WB failures even in the absence of halogens.

The presented data confirms that the rate of WB failures in PEMs increases in humid environments. Unfortunately, our results are not sufficient to estimate moisture acceleration factors in decapsulated devices. However, for devices encapsulated in plastics, the times to failure are much shorter than for decapsulated parts, and at 150 °C the accelerating factor of 85% RH can be estimated as 2.5 to 3.5 for OCN, to ~20 for BP MCs. This acceleration is likely due to a direct oxidation effect of moisture and to the enhanced transportation of liberated halogens [4, 15, 25] caused by moisture-induced plasticizing of epoxy materials.

## Conclusion

- Considering encapsulating molding compound as an environmental factor, the results of this work show that all environments, including oxygen, moisture, and MC, substantially decrease time to catastrophic Au/Al WB failure compared to vacuum conditions.
- The presence of MC during HTS decreases the characteristic life of WBs by a factor of ~10 in vacuum (at 198 °C) and by a factor of ~2 in air (198 °C). In humid environments (150 °C/85%RH) this decrease was estimated as a factor of ~3 to 5 for OCN and ~20 for BP MCs. This means that WBs in PEMs are especially vulnerable to possible manufacturing defects, thus requiring additional attention when commercial PEMs are to be qualified for high-reliability applications.
- HTS of PEMs in vacuum increases the characteristic life of WBs compared to air conditions on 5% to 35% only. However, for decapsulated devices, these variations are much more significant and at 198 °C time to the first WB failure increases in vacuum to more than 2500 hrs compared to ~250 hrs in air. This effect is due to the thermal decomposition of MC, which

provides a sufficient amount of oxidative agents to oxidize Au/Al intermetallics in PEMs and cause failures even in vacuum conditions.

- Characteristic times of WB failures in PEMs stored in air at temperatures from 190 °C to 200 °C vary from 500 to 1000 hrs and their activation energies are 0.71 to 0.79 eV for OCN MC and 0.91 eV for parts in BP MC.
- WBs failed in decapsulated devices after HTS in vacuum separated along the intermetallic/barrier metal interface, whereas in all other environments the separation occurred along the intermetallics exhibiting a grainy structure of a gold-rich composition with a grain size of 100 to 400 nm.

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